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**A COMPARISON OF THE EFFECTS OF SULFONATION ON SCREEN REJECTS
FROM STONE GROUNDWOOD AND THERMOMECHANICAL PULPS**

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THERMOMECHANICAL PULPS**

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ABSTRACT

The purposes of this laboratory study were to assess the effects of varying the level of sulfonation of stone groundwood screen rejects (SGWR) and thermomechanical pulp screen rejects (TMPR) before refining, to compare the properties of chemimechanical pulps from the two furnishes, and to gain some insight into the mechanisms involved. Rejects of both types from the same mill were sulfonated, refined and compared with respect to fiber and handsheet properties. Optimum strengthening occurred at a sulfonate content of about 1.75% and could be attributed to improved fiber conformability, fiber length preservation and surface area development. Relative to TMPR, SGWR consumes more chemical, gives slightly lower yield and is readily refined to low freeness with good surface development. It gives sheets of higher tensile strength, lower tear and zero-span tensile strength, and similar optical properties.

LITERATURE REVIEW

The last decade has seen the introduction of a variety of processes for combining the application of chemicals with mechanical pulping in disk refiners to improve pulp properties (1). Early in the development of these processes, it was recognized that application of chemicals before mechanical refining was complete resulted in generally better pulp strength, especially tearing resistance, than when fully refined pulps were subjected to chemical posttreatment (2). This led to interstage treatments and chip pretreatments. Pretreatments result in the cleanest fiber separation, with a minimum of fiber length reduction, and give the highest tear values. High levels of bonding are also achieved as a result of lignin softening, associated increases in fiber conformability and probably also increased bonding activity of the fines fraction. Although these factors have a beneficial impact on tensile strength and similar properties, they reduce the amount of light-scattering free surface within the sheet and opacity suffers.

Interstage treatments also improve fiber conformability (3), but differ from pretreatments in two major respects. First, because they are applied after the fiberization step they do not have as great an effect on long fiber content, tear strength or debris levels. Secondly, at least for sulfonation, the interstage treatment produces short fiber fractions of much higher specific surface than in the case of the corresponding pretreatment (4). This results in improved opacity and wet web properties, as well as reduced linting propensity.

Another way to reduce the loss of opacity associated with chemical treatment is to treat only

the long fiber fraction of the pulp. An additional advantage is that the chemical is directed to where it will do the most good and the chemical wastage and other detrimental effects associated with treating the finer fractions are avoided. This seems to have been first recognized by Lindholm (5), who later demonstrated the soundness of the principle by treating the long fiber fraction of stone groundwood (SGW) with chlorine dioxide before recombining it with the rest of the pulp (6). In subsequent work by Gummerus and Virkola (7), this treatment was applied to thermomechanical pulp (TMP) screen rejects with good results at high levels of chemical application.

Although chlorine dioxide may not be the chemical of choice, this mode of treatment has much to recommend it. Screen rejects represent a readily available stream of long fibers which, especially in TMP, are much in need of enhancement of their bonding potential. Furthermore, treating them separately avoids the detrimental effects of chemical consumption by the fines fraction, which already possesses sufficient bonding potential, allowing it to retain its opacifying power.

Instead of delignifying screen rejects as Lindholm did, Heitner *et al.*, chemically modified them with hydrogen peroxide and ozone. Applying this treatment before refining at fixed specific energy increased the specific surface area and specific volume of all fractions, increased the content of material passing a 100 mesh screen and slightly decreased the long fiber content. These changes combined to produce increased density, tensile strength and wet web properties, with no improvement in tear.

Sulfonation has emerged as the dominant chemical treatment in chemimechanical pulping, and its application to mechanical pulp screen rejects has accordingly been the subject of several recent studies. Gummerus (9) sulfonated TMP rejects before and after refining and rebled them with the parent pulp to produce pulps of improved strength with little loss in scattering coefficient. Shaw (10) treated TMP rejects from 5 mills with sodium sulfite before refining and demonstrated varying degrees of superiority of the resulting pulps over those that were refined without prior sulfonation.

Effects of sulfonation on both fiber properties and sheet properties were the object of a study by Heitner, Karnis and Attack (11). They treated refiner mechanical pulp screen and hydrocyclone rejects as well as stone groundwood (SGW) screen rejects with sodium sulfite to reach a sulfonate group content of 1.5-1.6% at a yield of 96-97%. This resulted in increases in fiber flexibility and conformability, together with a small increase in specific surface area. In subsequent refining the treated pulps developed specific surface more rapidly than controls, almost exclusively as a result of changes in the response of the short fiber (100/200 mesh) fraction. No effect of the chemical treatment on fiber length distribution after refining was observed. After refining at a given specific energy the treated pulps had lower freeness, linting propensity and shive content, together with higher wet web and dry tensile

properties. Tear at a given breaking length was unaffected.

Lindholm and Gummerus (12) compared the long fiber fractions of pressurized groundwood (PGW), SGW and thermomechanical pulp (TMP) with respect to their response to sulfonation and ozone treatment, with no subsequent refining. More recent work has concentrated on TMP rejects. Gummerus et al. (13) showed that at a given total specific energy consumption, increasing the degree of sulfonation before refining increases sheet density and tensile strength, decreases freeness and has no effect on fiber length distribution or tear. At a given freeness, long fiber content and average fiber length are increased, while fines content is decreased. Gummerus later studied the response of individual fiber fractions during the refining of whole sulfonated rejects (14). The finer fractions developed specific surface more rapidly, but bonding potential improved more rapidly in the coarse fractions.

PURPOSE AND SCOPE OF THE STUDY

Mills considering rejects sulfonation for improved mechanical pulp properties and reduction of the chemical pulp content in mixed furnishes are faced with a number of options. Typically, these will differ with respect to the level of sulfonation employed and whether the rejects to be treated are from a TMP or SGW line. The present study was undertaken to provide information that will assist in choosing among these options and to gain some insight into the mechanisms involved.

Rejects from SGW and TMP lines in the same mill were sulfonated and refined under laboratory conditions. The resulting pulps were characterized by measurements of fiber and handsheet properties.

RESULTS AND DISCUSSION

Properties of the Untreated Rejects

Table 1 characterizes the SGW and TMP rejects fractions (SGWR and TMPR) which were the raw materials for the sulfonation experiments. Both were obtained from a Finnish mill pulping Norway spruce (*Picea abies*). In both cases, the reject rate was 20%. The SGW rejects had lower freeness, higher specific surface, shorter fibers and higher shive content than the TMP rejects. The light scattering coefficient of the SGW rejects was higher as a result of its higher specific surface. Microscopic examination confirmed that its fiber surfaces were more extensively fibrillated than those of the TMP rejects.

Sulfonation

As shown in Fig. 1, SGWR consumed more chemical than TMPR at any given level of chemical application. If diffusion is assumed to be an important resistance in the sulfonation process, this can be attributed to the higher accessible surface area of SGWR. Alternatively, it may be an indication of different lignin reactivities or extractives contents.

Surprisingly, the TMPR gave pulps of higher sulfonate contents, as shown in Fig. 2. After

sulfonation of the SGWR, a smaller fraction of the sulfite consumed appeared in the pulp as sulfonate groups. This observation, together with the lower yields of SGWR after sulfonation, indicates that the TMPR lignin is less reactive, perhaps as a result of thermally induced condensation. The greater reactivity and smaller degree of condensation of the SGWR lignin allows more of it to be sulfonated to the point of solubility and removed by the pulping liquor, along with its sulfonate groups. More lignin, of equal or greater sulfonate content is retained by the TMPR.

Table 1 Properties of the rejects fractions before sulfonation.

	SGWR	TMPR
Cdn. std. freeness, mL	510	570
Shives, %	9.1	2.0
Length wtd. av. fiber length, mm	1.9	2.2
Bauer-McNett classification, %		
R28	54.2	53.9
R48/P28	13.4	19.6
R100/P48	9.7	9.7
R200/P100	5.4	2.9
R200	17.3	13.9
Specific surface area, m ² /g	1.83	1.42
Specific volume, cm ³ /g	3.14	2.92
Light scattering coefficient, cm ² /g	397	353
Tensile index, Nm/g	20.7	21.4
Tear index, mN m ² /g	5.9	5.2
Brightness	52.2	47.5

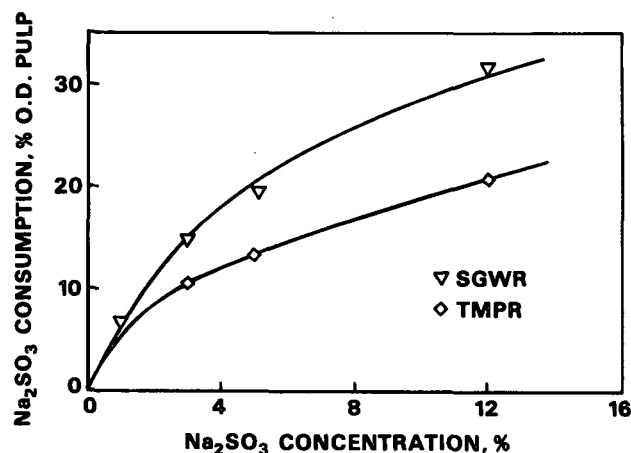


Fig. 1. SGWR consumes more Na₂SO₃ than TMPR when the amounts applied are equal. The rejects fractions were treated with Na₂SO₃ at 10% consistency.

Pulp Properties after Refining

Samples of both the untreated and sulfonated pulps were subjected to refining in the PFI mill at a constant level (3000 revolutions). The resulting pulps were characterized with respect to fiber and sheet properties to give the results shown in Tables 2 and 3.

Bauer-McNett classification. The size of the fraction retained on a 28 mesh screen (R28 fraction) underwent a sharp decrease when the pulp was

refined in the PFI mill, indicating that fibers were shortened as a result of the refining treatment. The degree of fiber shortening was progressively reduced by increasing degrees of sulfonation of the rejects before refining (Fig. 3 and 4). At the highest sulfonation level, the classification of refined SGWR was nearly indistinguishable from that of unrefined material. In the case of TMPR, the observation was similar, except that refining of the sulfonated pulp slightly increased the size of the fines fraction, at the expense of the middle fractions.

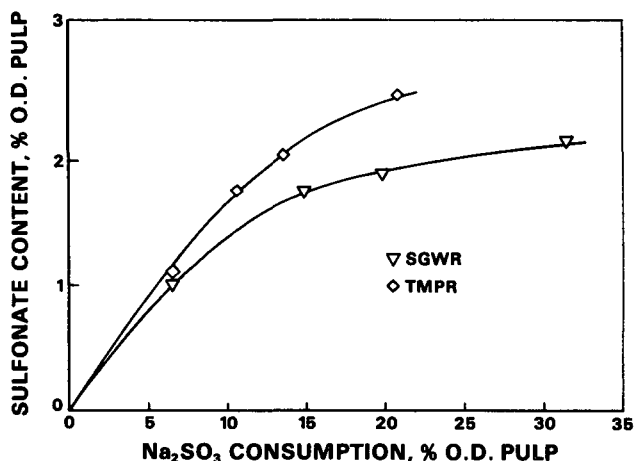


Fig. 2. Relative to SGWR, more of the sulfite consumed by TMPR is retained by the pulp as bound sulfur.

These observations may be interpreted in terms of lignin softening by sulfonation and its influence

on the mechanical properties of the fiber wall. Originally brittle and susceptible to breakage during refining, the lignified fibers of both pulp types become resilient and resist breakage. The slightly greater degree of fines generation during sulfonated TMPR refining may be a consequence of the lower rate of primary fines generation during the earlier refining stages.

It has previously been observed that rejects sulfonation prior to refining does not affect fiber length distribution after refining to a given specific energy consumption (11,13). In addition, it has been shown that fiber length is preserved when refining to a given freeness, since sulfonation decreases the energy consumption at constant freeness (13). The present study shows that fiber length is also preserved when refining is done at a constant level of mechanical action. It may be inferred that energy is less readily transferred to the pulp after sulfonation, and that SGWR and TMPR differ little in this regard.

Specific surface and freeness. Sulfonation resulted in a slight increase in the specific surface of the unrefined pulps, as illustrated in Fig. 5. Upon subsequent refining, the increase in specific surface was slightly greater for sulfonated than for unsulfonated pulps. SGWR underwent a larger increase in surface during refining, and the effect of sulfonation on the increase was greater than for TMPR. The higher initial shive content of the SGWR may account in part for its greater response, but it is probably mainly due to increased surface development in the short fiber fraction (11,14). Note that sulfonation enhanced surface development in spite of decreased fines generation.

Table 2 Fiber properties.

Pulp	Na ₂ SO ₃ Conc., %	Consump. of Sulfonate		Yield, %	PFI Refining, 3000 rev.	Freeness, mL	Bauer-McNett Classification, %					Specific Surface Area, m ² /g	Specific Volume, cm ³ /g
		Na ₂ SO ₃ % on o.d. pulp	Content, %				R28	R48/P28	R100/P48	R200/P100	P200		
SGWR	0	0	0.02	100.0	-	510	54.2	13.4	9.7	5.4	17.3	1.82	3.14
"	1	6.5	1.01	93.3	-	--	--	--	--	--	--	1.76	3.08
"	3	14.8	1.73	93.3	-	--	--	--	--	--	--	1.90	3.16
"	5	19.7	1.88	89.9	-	--	--	--	--	--	--	2.01	3.00
"	12	31.3	2.15	89.9	-	--	--	--	--	--	--	2.02	3.02
"	0	0	--	100.0	+	275	38.4	20.6	13.3	7.2	20.5	2.75	2.91
"	1	6.5	--	93.3	+	207	50.7	16.1	11.7	6.2	15.3	2.66	3.11
"	3	14.8	--	93.3	+	196	52.1	14.3	10.8	5.9	16.9	2.97	3.14
"	5	19.7	--	89.9	+	188	52.4	14.3	10.5	5.2	17.6	3.23	3.20
"	12	31.3	--	89.9	+	185	53.1	14.4	10.5	5.8	16.2	3.35	3.12
TMPR	0	0	0.01	100.0	-	570	53.9	19.6	9.7	2.9	13.9	1.42	2.92
"	1	6.6	1.10	94.8	-	--	--	--	--	--	--	1.46	3.16
"	3	10.6	1.77	93.8	-	--	--	--	--	--	--	1.54	2.91
"	5	13.4	2.04	92.2	-	--	--	--	--	--	--	1.53	2.96
"	12	20.8	2.51	91.2	-	--	--	--	--	--	--	1.63	2.89
"	0	0	--	100.0	+	440	42.9	22.2	12.5	4.2	18.2	1.78	3.02
"	1	6.6	--	94.8	+	345	50.1	20.0	10.7	4.0	15.2	1.96	3.10
"	3	10.6	--	93.8	+	335	53.1	18.1	8.2	3.2	17.4	1.96	2.99
"	5	13.4	--	92.2	+	330	54.1	17.4	8.0	2.9	17.6	2.06	2.99
"	12	20.8	--	91.2	+	330	54.0	17.3	8.2	3.0	17.5	2.08	2.91

Table 3 Handsheet properties.

Pulp	Na ₂ SO ₃ Conc., %	Wet Mat Density, g/cm ³	IPC Density, g/cm ³	Tear Index, mN m ² /g	Tensile Index, Nm/g	Tensile Stiffness, KN/m	Elastic Modulus, GPa	Tensile Stretch at Zero-span			TAPPI Bright- ness, %	Light Scatter- ing Coeff., cm ² /g
								Energy, Abs., J/m	Break, %	Breaking Length, km		
SGWR	0	0.149	0.307	5.86	20.8	140	0.752	10.92	1.4	8.20	52.2	397
"	1	0.160	0.353	5.97	24.5	189	1.161	11.46	1.2	8.39	45.9	360
"	3	0.171	0.392	6.04	34.1	223	1.552	20.86	1.7	9.32	45.5	328
"	5	0.176	0.425	6.28	35.2	245	1.834	19.06	1.5	10.04	48.4	330
"	12	0.183	0.450	6.55	35.2	272	2.136	17.57	1.3	10.14	48.0	325
"	0	0.168	0.363	4.87	30.8	185	1.236	18.08	1.7	8.60	48.5	404
"	1	0.176	0.471	5.37	47.8	306	2.515	31.44	1.8	10.06	43.8	314
"	3	0.184	0.542	5.37	54.1	350	3.391	31.70	1.6	10.80	44.3	280
"	5	0.186	0.543	5.08	57.9	350	3.357	42.76	1.9	11.04	44.5	276
"	12	0.188	0.572	5.23	58.4	350	3.515	40.54	1.8	10.96	44.9	266
TMPR	0	0.151	0.274	5.18	21.4	123	0.580	13.79	1.7	8.75	47.5	353
"	1	0.155	0.326	5.99	28.8	182	1.019	21.07	1.9	9.01	42.6	320
"	3	0.178	0.339	6.28	34.5	204	1.222	27.17	2.0	9.03	43.2	299
"	5	0.180	0.382	6.45	36.1	213	1.447	28.60	2.0	10.17	45.7	304
"	12	0.187	0.381	6.51	37.6	223	1.523	30.85	2.1	10.06	47.4	310
"	0	0.163	0.308	4.84	27.9	169	0.923	18.34	1.7	8.80	46.9	359
"	1	0.173	0.417	6.05	45.4	258	1.893	38.17	2.1	10.55	42.3	305
"	3	0.185	0.462	6.23	46.6	288	2.354	31.93	1.8	10.76	44.0	284
"	5	0.190	0.481	6.24	48.9	288	2.481	41.25	2.2	10.94	44.5	280
"	12	0.194	0.500	6.20	50.0	288	2.582	43.54	2.2	11.40	45.4	277

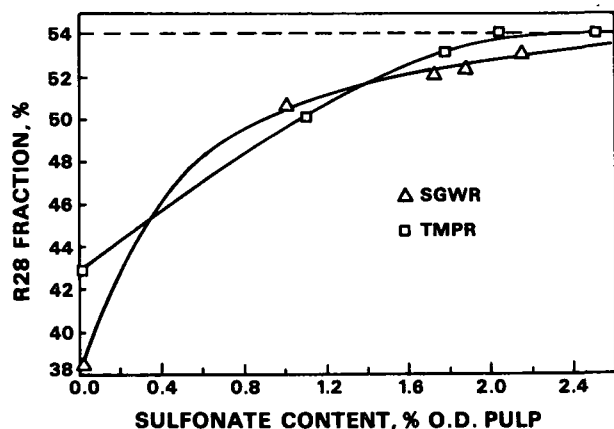


Fig. 3. With increasing degree of sulfonation, the size of the long fiber fraction in the refined pulps approaches that in the unreformed rejects (dashed line).

The freeness after refining was decreased by sulfonation, as is to be expected on the basis of the effects on specific surface discussed above. Improved fiber flexibility may also have contributed to the freeness decrease. SGWR freeness was more sensitive to refining and to increased degree of sulfonation; this may be due to greater disruption of the fiber wall relative to the more intact TMPR fibers.

Specific volume and fiber conformability. Although improved fiber conformability is often associated with increased swelling of the fiber wall, there

was no significant effect of sulfonation on hydrodynamic specific volume before or after refining. As has been pointed out by Attack (15), this is not inconsistent with the occurrence of "inward swelling" as might be expected if the primary wall were to remain intact. An intact primary wall would restrict outward swelling, which would otherwise be detected by measurements of filtration resistance.

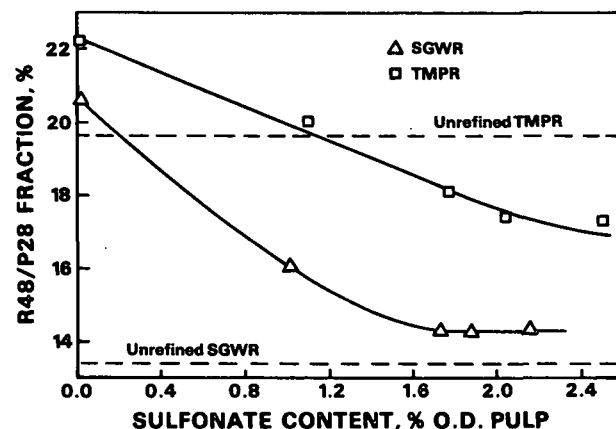


Fig. 4. The size of the R48/P28 fraction, which contains a large proportion of broken fibers, decreases with increasing degree of sulfonation. In TMPR, refining converts a part of this fraction to fines.

Further indications of fiber conformability are provided by measurements of wet mat density and dry sheet density. Figure 6 illustrates the effects of sulfonation and refining on wet mat density; both

effects are positive, but the effect of refining decreases with increasing degree of sulfonation. This suggests that wet conformability can be substantially achieved by sulfonation alone, and subsequent refining adds little unless sulfonation is incomplete. The two pulps behave similarly in this respect, although there is a difference between the forms of their respective response curves.

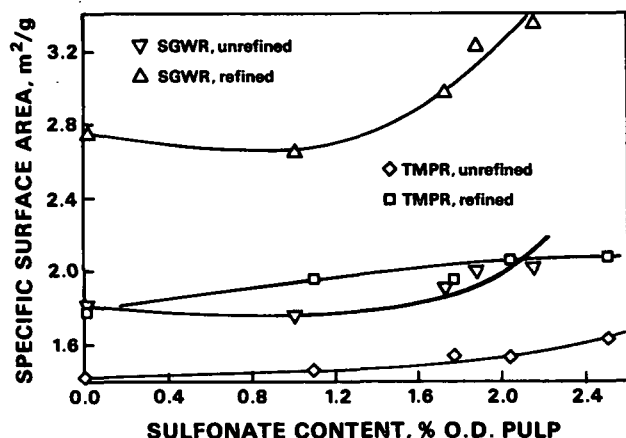


Fig. 5. Sulfonation increases specific surface and its response to refining more for SGWR than TMPR.

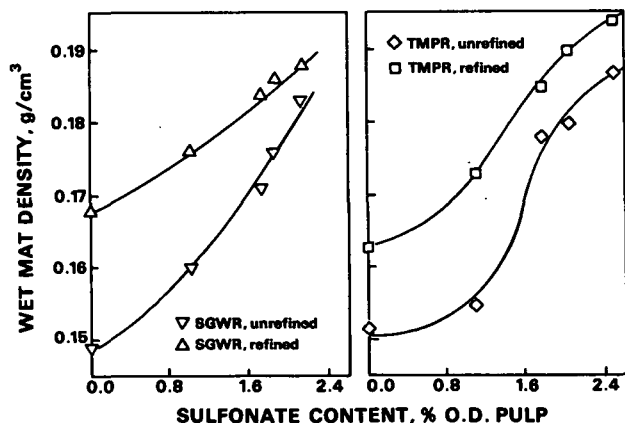


Fig. 6. Wet mat density is increased by sulfonation and by refining. The effect of refining is smaller at high sulfonate contents.

The density of TAPPI handsheets, as determined by the standard IPC rubber platen caliper gage, is plotted in Fig. 7. Sulfonation increases sheet density and improves its response to refining. This means that the effect of refining on dry sheet density, unlike that on wet mat density, increases with increasing degree of sulfonation. Sulfonation alone is enough to facilitate the limited collapse achievable by water swollen cell walls, but the more complete degree of collapse necessary for good bonding in the dry state can only be achieved by physical disruption of the wall structure. The latter is promoted by sulfonation, perhaps through enhanced accessibility of the internal wall structure to water.

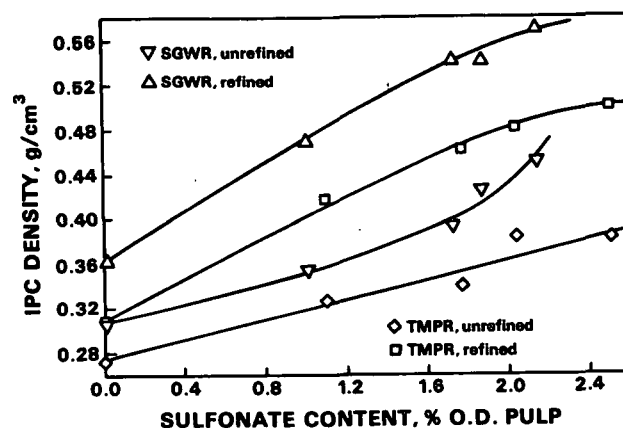


Fig. 7. Sulfonation increases sheet density and improves its response to refining.

Tensile strength and modulus. The tensile strength of SGWR was increased more by refining than that of TMPR, and its response was more effectively enhanced by sulfonation, as shown in Fig. 8. These effects paralleled the corresponding effects on sheet density discussed above. Figure 9 illustrates the close association between density and tensile strength of all of the experimental pulps, but also shows that the tensile strength of the unrefined SGWR pulps is somewhat lower than expected on the basis of sheet density. This is believed to be due to their high content of shives, which give rise to failure-initiating stress concentrations within the sheet. The better correlation between elastic modulus (a prefailure phenomenon) and tensile strength shown in Fig. 10 lends support to this argument.

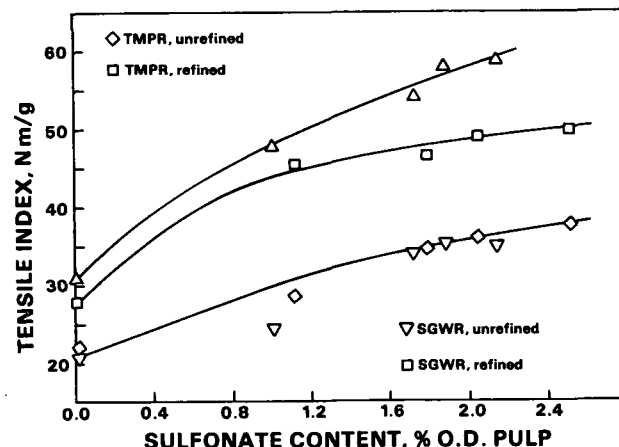


Fig. 8. Tensile strength and its response to refining were enhanced by sulfonation, especially for SGWR.

Tear index. As shown in Fig. 11, the tear-tensile relationships of the sulfonated but unrefined SGWR and TMPR were similar. Upon refining, however, the greater tensile strength increase of the SGWR was accompanied by a disproportionate loss of tear strength. As a result, the tear index of sulfonated and refined TMPR was considerably higher than that of the corresponding SGWR at any given tensile

strength, although SGWR could be raised to higher tensile strength values.

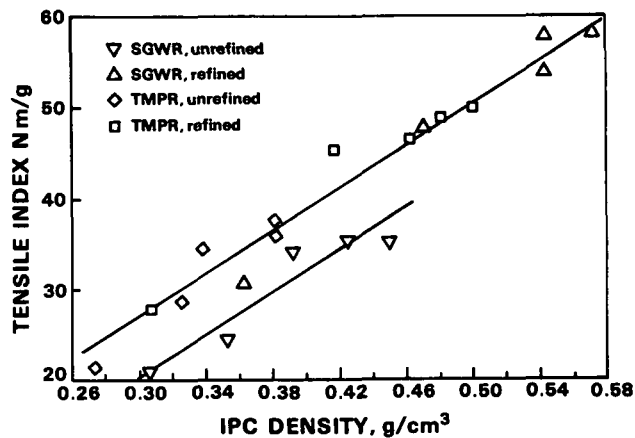


Fig. 9. The tensile strength of unrefined SGWR is lower than expected, presumably as a result of shive-initiated failure.

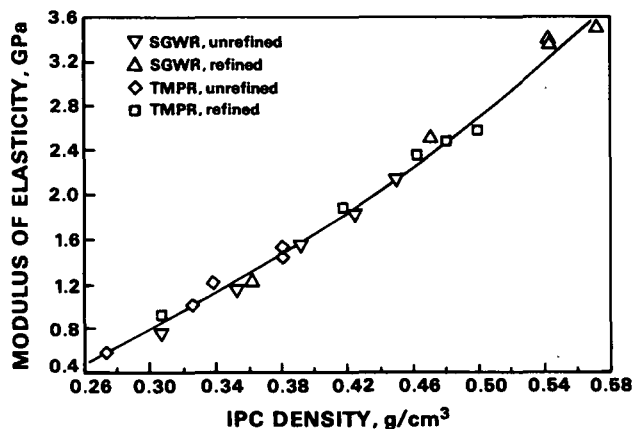


Fig. 10. The effect of sulfonation and refining on elastic modulus parallels the corresponding effects on sheet density.

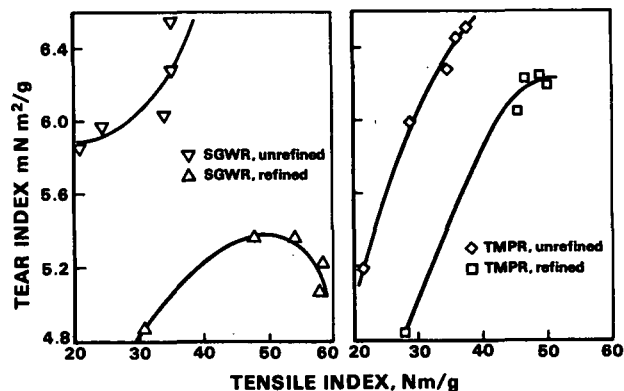


Fig. 11. Upon refining, SGWR undergoes a greater loss in tear than TMPR.

A probable reason for the inferior tear strength of sulfonated and refined SGWR is lower fiber strength. Figure 12 shows that the zero-span strength of TMPR is higher at any given sheet density, suggesting that the average strength of its fibers is greater, presumably as a result of a lower level of damage during fiber separation.

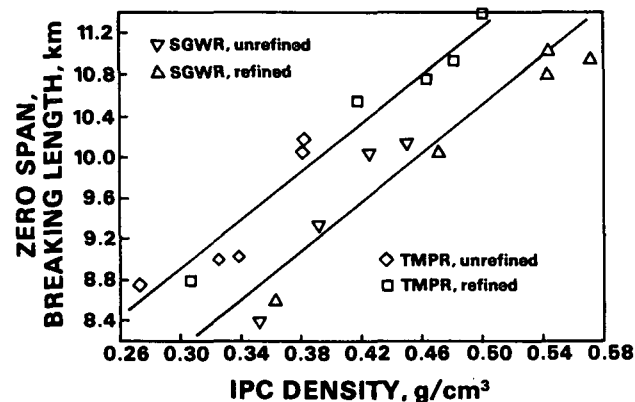


Fig. 12. At a given sheet density, TMPR exhibits higher zero-span tensile strength than SGWR, suggesting that its fibers are stronger.

Optical properties. The light scattering power of both pulp types was decreased by sulfonation, as shown in Fig. 13. The initial advantage possessed by SGWR persisted through sulfonation and refining. The scattering coefficients of refined pulps were higher than those of unrefined pulps having the same tensile strength (and higher sulfonate content). However, comparisons within individual pairs of data points show that refining decreased the scattering coefficients of all pulps except the unsulfonated controls. Apparently, the decrease in free surface due to improved bonding is greater than the surface generated during refining.

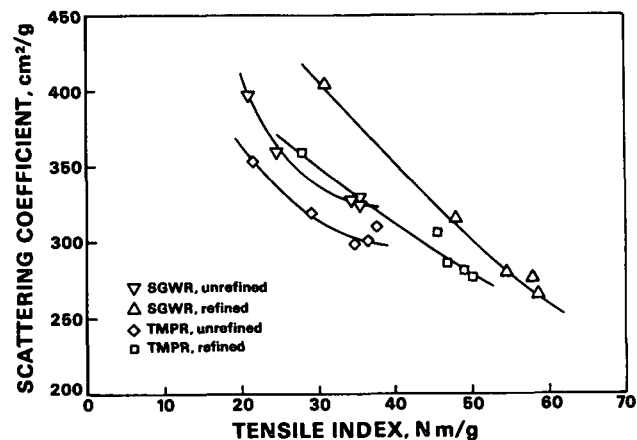


Fig. 13. Sulfonation increases tensile index at the expense of scattering power.

As shown in Fig. 14, sulfonation reduced pulp brightness by 0-8 points, the effect being greatest for low-level sulfonation of SGWR and least for

high-level sulfonation of TMPR. In both cases, the brightness went through a minimum as the sulfonate content was increased. Refining tended to reduce the brightness, especially in the case of SGWR. These changes suggest that the main effect on brightness arises from the loss of light scattering power, with some chromophore reduction at the higher levels of sulfonation.

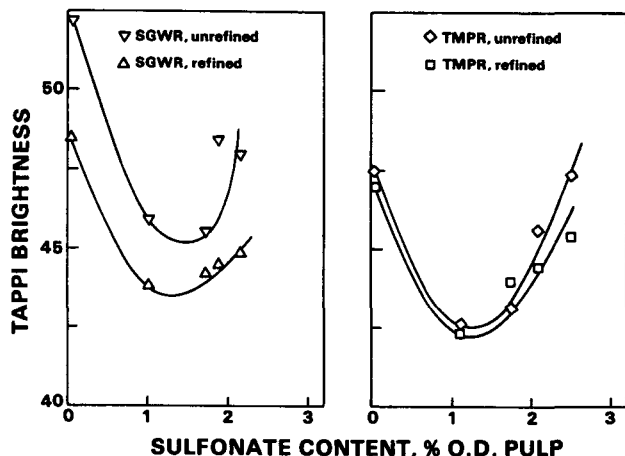


Fig. 14. Brightness is first decreased and then increased as the degree of sulfonation is increased.

EXPERIMENTAL

Sulfonation

Rejects were sulfonated at 10% consistency in solutions containing 1, 3, 5 or 12% Na_2SO_3 . The initial pH was adjusted to 8.0 with H_2SO_4 and fell during the treatment by 0.1 to 1.7 units, depending on the Na_2SO_3 concentration. The treatments were performed in microdigesters which were continuously agitated in a thermostatically treated oil bath. The bath temperature was increased from 80 to 150°C in 85 minutes and maintained at 150°C for 120 minutes.

After the sulfonation treatment, the residual Na_2SO_3 concentration was determined iodometrically by the method of Palmrose (16). After being washed, the pulps were refined at 10% consistency in a PFI mill for 3000 revolutions.

Pulp properties. Latency was removed by disintegrating the pulps for 10 minutes at 90°C in a TAPPI disintegrator. TAPPI test methods were used to test handsheets of the treated pulps. Freeness was determined according to T227 and sheets were formed according to T205, except that white water recirculation was employed, and the sheets were dried between a felt and a heated cylinder.

The physical properties of the handsheets were determined according to T220. Caliper was determined by the IPC rubber platen caliper gage (17). Light scattering coefficient and brightness were determined according to T425 and T452, respectively.

Hydrodynamic specific surface and specific volume were determined by the constant rate filtration method described by Ingmanson et al. (18).

Shive content was determined by a vibratory plate screen with 0.006-inch slots. Fiber classifications were performed in a four-compartment Bauer-McNett classifier according to T233.

SUMMARY AND CONCLUSIONS

Table 4 summarizes the effects of sulfonation on rejects properties and compares the properties of pulps from the two rejects types.

Table 4 A summarizing comparison of mechanical and chemimechanical pulps from rejects after 3000 PFI mill revolutions.

	SGWR		TMPR	
Na_2SO_3 consumed, % o.d. wood	0	14.8	0	10.6
SO_3H content, % pulp	0	1.73	0	1.77
Yield, % o.d. rejects	100	93.3	100	93.8
L-factor	59	67	65	71
Cdn. std. freeness, mL	275	196	440	335
IPC density, g/cm ³	0.36	0.54	0.31	0.46
Tensile index, Nm/g	31	54	28	47
Tear index, mN m ² /g	4.9	5.4	4.8	6.2
Scattering coefficient, cm ² /g	400	280	360	280

Both thermomechanical pulp screen rejects (TMPR) and stone groundwood rejects (SGWR) can be sulfonated before refining to substantially improve their properties. The strength improvement comes about as a result of improved fiber conformability, fiber length preservation and surface area development. The sulfonate content of the treated pulps should be approximately 1.75%, a level which corresponds to consumption of 15% Na_2SO_3 by SGWR or 11% by TMPR.

Relative to TMPR, SGWR consumes more chemical, gives a slightly lower yield and is readily refined to low freeness with good surface development. The resulting pulp gives sheets of higher density and tensile strength but lower tearing resistance. The brightnesses and light scattering coefficients of the two pulps are similar. At a given sheet density, zero-span tensile strength is lower for SGWR than TMPR, suggesting that fiber strength contributes to the lower tear of the former.

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